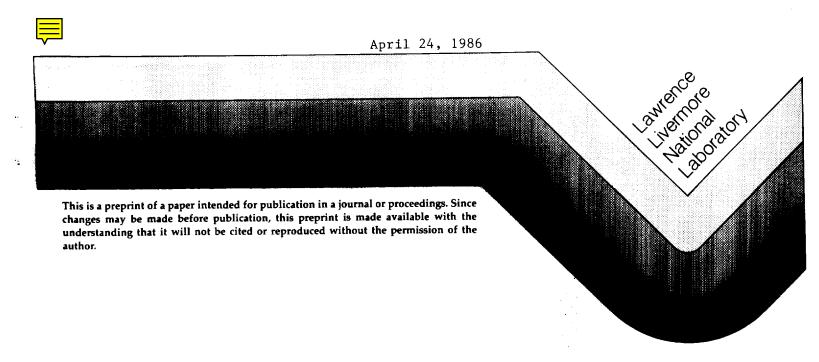
Practical Method of Preparing Precision, Ultra-Fine Sapphire Surfaces

Eberhard Prochnow David F. Edwards

CIRCULATION COPY SUBJECT TO RECALL IN TWO WEEKS

Optical Sciences and Engineering Group Lawrence Livermore National Laboratory

Applied Optics



## DISCLAIMER

This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor the University of California nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial products, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement recommendation, or favoring of the United States Government or the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or the University of California, and shall not be used for advertising or product endorsement purposes.

## PRACTICAL METHOD FOR PREPARING PRECISION, ULTRA-FINE SAPPHIRE SURFACES

Eberhard Prochnow and David F. Edwards
Optical Sciences and Engineering Group
Lawrence Livermore National Laboratory
Livermore, CA 94550

A precision surface has a high degree of conformity to a desired figure. An ultra-fine surface has a high degree of smoothness. Several authors (1,2) have described methods of preparing either a precision surface or an ultra-fine surface for sapphire. A few claim to be able to do both. In general, they describe methods suitable for quanitities of 100 or more pieces that require extensive fixturing and initial tooling. below is the method we use for preparing small quantities (5-10 pieces) of polished sapphire ( $Al_2O_3$ ) wafers having surface figure of  $\lambda/8$  or better and surface roughness of 2-3 Å RMS. elaborate fixturing or special machines are required, and the total time for completing one side of a block of seven is about 16-20 hours. The final polishing steps are done using colloidal silica on pitch. This is important in that it permits us to alter the figure of the surface as well as to achieve the smoother surfaces.

We concur with previous investigators (2) that the removal mechanism for the colloidal silica is primarily by chemical reaction. The polishing pitch, as used by us, adds to the chemical reaction of the colloidal silica with sapphire making figure control a possibility. As reported by others (2) the changing of surface figure using colloidal silica with cloth or Pellon pads is very slow and not practical. The faster chemical reaction of the pitch-colloidal silica increases the removal rate for the sapphire surface. This is important for preparing wafers having minimum or zero disturbed surface layers. During the grinding steps the surface is abraded by a complex series of microfractures and chipping. This leaves a finite layer of

<sup>\*</sup>Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract No. W-7405-ENG-48.

microscopic fractures propagating into the material a distance depending on the details of the operation. We have measured damage depths of a few microns to several hundred microns depending on the type of material (e.g. glass, crystal, etc.) and the removal process. This remnant of work damage, called the damage layer, must be removed before the sapphire surface is usable for some applications. We have reason to believe that pitch polished surfaces using colloidal silica is an efficient method of removing this damaged layer.

Given below are details of the procedures we have used to prepare damage free sapphire surfaces. The starting material was a  $60^{\circ}$  leuco-sapphire (undoped  $\mathrm{Al}_2\mathrm{O}_3$ ) rod 19 mm diameter and about 75 mm long. An inspection polish was first given to the outer diameter of the rod. We define an inspection-polished surface to have a shine but ignore small pits, small scratches and figure. To generate the inspection polish, the rod was lapped while mounted between centers on a tool grinding machine (3). Starting with 400 grit boron carbide in water and finishing with 3 micron diamond in water the cylinder inspection polish was completed in about 8 hours.

Wafers 3.3 mm thick were cut from the rod using a diamond cut-off saw. The edges of each wafer were beveled and inspection polished using diamond dust to reduce edge flaking or breaking that might cause scratching during later steps. The optimum bevel angle was found to be  $60^{\circ}$  relative to the rod axis.

All the steps that follow were made with the parts cemented  $^{(4)}$  to a 64 mm dia. x 12.7 mm pyrex disk. One wafer was at the center surrounded by six equally spaced wafers. An aluminum disk could also be used in place of the pyrex disk. An advantage of pyrex is its transparency which permits monitoring wafer parallelism during the fabrication process.

With the seven wafers cemented to the pyrex disk they are first "touched" using a diamond wheel of a No. 11 Blanchard grinder to correct variations in wafer thickness. For the

Blanchard step we use a 6.4 mm wide wheel with 100 grit diamond with 75% concentration in a resin bond. A water soluble detergent (5) was used as the coolant. The Blanchard step, like the sawing step, can produce an excessive damage layer, so care must be exercised to avoid over aggressive stock removal. We first removed about 0.15 mm from all seven wafers at a rate of 0.23 mm/min. This was followed by removing 0.03 mm at a rate of 0.08 mm/min. with a 5 min. "spark out" (i.e., continue the Blanchard with no wheel advance).

The grinding steps were done using a hand grinder  $^{(5)}$ . Starting with a cast iron lap and 400 grit boron carbide abrasive in water we remove about .05 mm of material. This was followed by a 3  $\mu$ m diamond dust in distilled water on a copper KEMET lap  $^{(6)}$ . About 0.03 to 0.04 mm was removed with this step. These two grinding steps, cast iron and copper Kemet, were completed in about 15 minutes each.

After brushing and cleaning the copper Kemet lap we use  $1\mu$  diamond dust in distilled water to bring the surface to about one wave convex across the entire block. At this step the surface finish, as measured using the Optical Heterodyne Profilometer (7), was 50-70 Å RMS. The 1  $\mu m$  diamond step required about 30 minutes.

The polishing was performed using a pitch  $lap^{(8)}$  on a bowl feed polisher  $^{(9)}$ . The lap, 75 mm diameter with V-grooves spaced 12 mm, rotated at 250 rpm. The work was on top and oscillating at 32 strokes per minute. The position of the work relative to lap center was operator controlled to obtain the desired figure. The pressure on the work was approximately 500 gm/cm². For this polishing step a 50% (by volume) distilled water - 50% colloidal silica  $^{(10)}$  (60  $\mu$ m grit size) slurry was constantly applied to the center of the lap. After about 15 minutes the surface roughness had been reduced to about 10-12 Å RMS and the figure was about 1/2 wave convex for each wafer. A second 15 minute cycle with no change in the polishing conditions produced a 7-8 Å RMS surface with the center piece flat to about 1/8 wave

convex. The six outer pieces had a slight turned down edge. Following a third 15 minute cycle again with no change in the polishing conditions the surface finish of all seven parts was 2-3 Å RMS and the average figure for each piece was 1/2 wave. The figure of the center piece was better than 1/8 wave, and the six outer pieces were better than one wave.

Starting with the rough ground rod about 16 to 20 hours were required to polish one side of the 7 wafers. Polishing the second side of the 7 wafers to this same figure and smoothness would require another 3-4 hours.

We believe the described preparation procedure to be practical in that no elaborate or expensive fixturing or machinery was required. The procedure is best suited for small quantities. The technique could probably be scaled up for preparing polished disks of larger diameters in lots of seven. Also the surface-damage layer is believed to be negligibly small because of the chemical action of the pitch-colloidal silica combination. For our application the sapphire disks were used as plane-parallel low scatter windows but the process is equally applicable for epitaxial silicon substrates.

We wish to acknowledge the advice and encouragement given us by the other members of the Optical Sciences and Engineering Group during the course of this work.

## REFERENCES

- Y. Namba and H. Tsuwa, Annals of the CIRP <u>26</u> (1) 325 (1977);
   A. Reisman, M. Berkenblit, J. Cuomo, and S. A. Chan, J. Electrochem Soc <u>118</u> (10) 1653 (1971);
   V. J. Lazazzero, Laser Focus, Aug. 1978, p. 75.
- 2. H. W. Gutsche and J. W. Moody, J. Electrochem Soc  $\underline{125}$  (1) 136 (1978).
- Universal Tool Grinder, Model ES, K.O. Lee Co., Aberdeen, S.
   Dak.
- 4. No Load Mounting Cement, Do All Co., Des Plaines, IL.
- 5. Model 6T, R. Howard Strasburgh, Inc., Huntington Beach, CA.
- 6. Engis Corp., Morton Grove, IL.
- 7. G. Sommargren, Appl. Opt. 20, 610 (1981).
- 8. Universal Shellac and Supply Co., Hicksville, NY.
- 9. Type M423, Automatic Bowl Feed Polisher, American Optical Co., Southbridge, MA.
- 10. No. 2360, Nalco Chemical Co., Oak Bridge, IL.